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NOTEBOOK NO. 4573
ISSUED TO M. Fourné
ON 1/11 1982
DEPARTMENT ADDITIVES RESEARCH
RETURNED 7/21 1988

PVC STABILIZERS

— SCIENTIFIC NOTEBOOK CO. —
5007 WEST DONNA DRIVE
STEVENSVILLE, MICHIGAN 49127

From Page N. _____

This experiment is designed in order to compare mercaptoethyl esters with other S-containing chemicals as thioextenders. Products directly available from the shelves were compared with MET at 0.3 phr level. CN 10302 (22.1%) is the tin stabilizer

Results.

- a) - standard pipe compound is used
- Roll Mill 5 min 350°F / Press Test 10 min at 350°F

Stabilizer	S. compound (0.30 phr)	WI	Test #
0.15 phr CN10302 /		50.2	28-1
"	Diisooctylthiodipropionate	51.9	28-2
	n-decylmercaptoacetate	27.3	28-3
	cyclohexylmercaptan	41.2	28-4
	2-MET	56.0	28-5
	Thiolaurylanthydride (TLA)	29.4	28-6

Except for the diisooctylthiodipropionate which has little effect (probably within experimental error) the other mercaptans and TLA have negative effects.

Only MET was clearly a synergist

- b) Oven test 400°F

The Oven test confirms visually the results of the presstest.

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This time CN10405 — $\text{BuSn}^{\text{III}}\text{SC}_{12}\text{H}_{25}$ — (29.1% Sn) reagent was chosen as the tin stabilizer — Sulfur containing materials were those used by COUGH in US 3,928,285 in the example of table III, page 13 except for benzylthioglycolate (not available).

- Standard pipe compound (210.8g)
- Roll Mill 5 min 350°F + 10 mm press test
- Brabender 200°C / 60 rpm / 68g charge.

Results

Test #	Fusion Time	Fusion Torque	Xlink Time	Xlink Torque	Stability Time	WI	0.15 phr CN10405 + S chemicals (0.30 phr)
1	1.1	2400	1650	6.2	5.1	24.1	—
2	0.7	2450	1610	5.6	4.9	39.9	Isooctylthioglycolate.
3	0.8	2460	1520	5.7	4.9	39.7	i-octyl β mercaptopropionate
4	0.8	2300	1520	5.7	4.9	28.0	octadecylthioglycolate (EVANS)
5	0.7	2490	1610	5.5	4.7	41.0	n-butylthioglycolate (EVANS)
6	0.9	2320	1620	5.9	5.0	25.9	Dodecylmercaptan (Philippi)
7	0.8	2320	1520	5.7	4.9	31.0	Octylmercaptan (FUKA)
8	0.8	2350	1510	5.3	4.5	29.2	Thiolaurylanhydride.
9	0.7	2410	1610	6.1	5.4	64.4	2-ME Pelargonate (4360-1)
10	0.8	2390	1600	6.0	5.2	59.9	2-ME-C10? (4490-25)

- Oven test confirms press test results.
- Brabender also indicates MEE superiority.

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The same S-containing chemicals including benzyl thioglycolate will be checked in combination with CN ~~8900~~ 8900 - Bu Sn (S) IOMA (29.0% tin content). This time in order to provide a variety of products Mercaptoethyl stearate and ME succinate will be used as examples of MEE

Results

- Standard pipe compound
- Roll milling 5 min 350°F / Press test 10 min 350°F

~~Stabilizer~~
Test #

(4573-31)	Stabilizer	WI
1	0.15 phr CN 8900 / —	27.7
2	" / isooctyl thioglycolate	35.6
3	" / isooctyl mercapto propionate	38.8
4	" octadecyl thioglycolate (EVANS)	29.0
5	" n-butyl thioglycolate (EVANS)	40.0
6	" dodecyl mercaptan (Philipps)	28.4
7	" octyl mercaptan (Fluka)	36.0
8	" Thiolaurylanthidine	38.0
9	" 2-MES (SNEA)	54.8
10	" ME-succinate (4310-40)	56.7
11	" Benzyl thioglycolate	49.7

Over test 400°F confirms superiority of MEE both in early color (5-10-15 min) and long term (less bubbles at 30 min)

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TITLE MEE Patent Examples

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Using the sample Sulfur containing materials at same concentration,
0.30 phr (see page 31) the tin material used is T137[®] (Sn = 14.5%)

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From Page No. 21

Using the same sulfur containing materials at same concentration (0.30phr) (see p 31) the tin material used is T137® (Sn = 14.5%) - Products are evaluated in a standard pipe compound formulation

- Roll Mill 5mm 350°F + 10mm Press Test 350°F

Test #	Stabilizer = 0.15phr T137	WI
(4573-33)	+ S-Chemicals:	
1	—	1.7
2	i-octyl mercapto acetate	0.4
3	i-octyl p-mercapto propionate	3.7
4	octadecyl mercapto acetate (Evans)	2.3
5	n-butyl mercapto acetate (Evans)	7.4
6	Dodecyl mercapton (Phillippo)	-8.7
7	Octyl mercapton (Fluka)	-6.5
8	Thiollauryl anhydride	16.4
9	2-MES (SNEA)	22.9
10	ME-Succinate (4310-40)	22.1
11	Benzyl thioglycolate	16.9

Despite a low tin level (half of the one used with CN 10405 and CN 10395) performance of MEE remains clearly better than those of other mercaptans and TLA

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Always using the same collection of sulfur compounds at the same concentration 0.30 phr (compare p 33), BTSA is checked. However BTSA is high in tin content (53.1%) and the concentration will be reduced from 0.15 phr to 0.10 phr to compensate.

- Standard pipe compound
- Roll Mill 5mm 350°F + Press Test 10 mm 350°F

Results

Test #	Stabilizer 0.10 phr BTSA + (4573) , 0.30 phr S-Chemicals	WI
34-1	to _____	30.3
34-2	Isocetyl thioglycolate	42.5
34-3	Isocetyl mercapto propionate	49.2
34-4	Octadecyl thioglycolate (Evans)	29.7
34-5	n-Butyl thioglycolate (Evans)	46.7
34-6	Dodecyl mercaptan (DDM)	39.8
34-7	Octyl mercaptan (Fluka)	42.9
34-8	Thiollaurylanhydride	44.5
34-9	2-MES (SNEA)	50.4
34-10	ME-Succinate (4310-40)	55.6
34-11	Benzyl thioglycolate	45.0

Here also we note the very clear superiority of MEE over other thiols and thiollaurylanhydride

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From Page No. 24

1,3-bis (4,4,6-trimethyl-1,3,2-dioxaborinyl-2-oxy) (tetra-n-butyl distannoxane) (Stabilizer A) was supplied by SNEA
Sn content is 31.0% Sn i.e. similar to CN 8900 and CN 10405.

Due to the small quantity available the product will be used only with best materials as suggested by Gough (US patent # 3,928,235, Ex # 20 to 27 (Table III, page 13) and Thiollaurylanhydride.

Results

- Standard pipe compound
- Roll Mill 5mm 350°F / Press Test 10mm 350°F

Test # Stabilizer = 0.15 phr of A +
(4573) 0.30 phr of S chemicals

		WI
35-1	—	deep purple / brown
35-2	Isa Butyl thioglycolate (Evans)	-7.5
35-3	i-octyl mercapto propionate	5.4
35-4	Thiollaurylanhydride	-25.9
35-5	Octylmercaptan (Fluka)	-10.8
35-6	Benzyl thioglycolate	10.9
35-7	i-octyl mercapto acetate	-20.2

- The MEE were not checked for lack of material (organotin berate) and the fact that we don't claim such products.
- In accordance with Gough's results (EX 12 and 27 p13) the organotin berate efficiency is close to zero
- Addition of any of the S chemicals does boost the color but performance remains low (compare with CN 10405 or CN 8900 at same Sn level). Even in the case of T137 (page 4573-33) with about half of the tin contents the performance is overall better

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